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**No. 21.—The Separation of Arsenic, Tin and
Antimony.**

BY

W. R. LANG, C. M. CARSON and J. C. MACKINTOSH.

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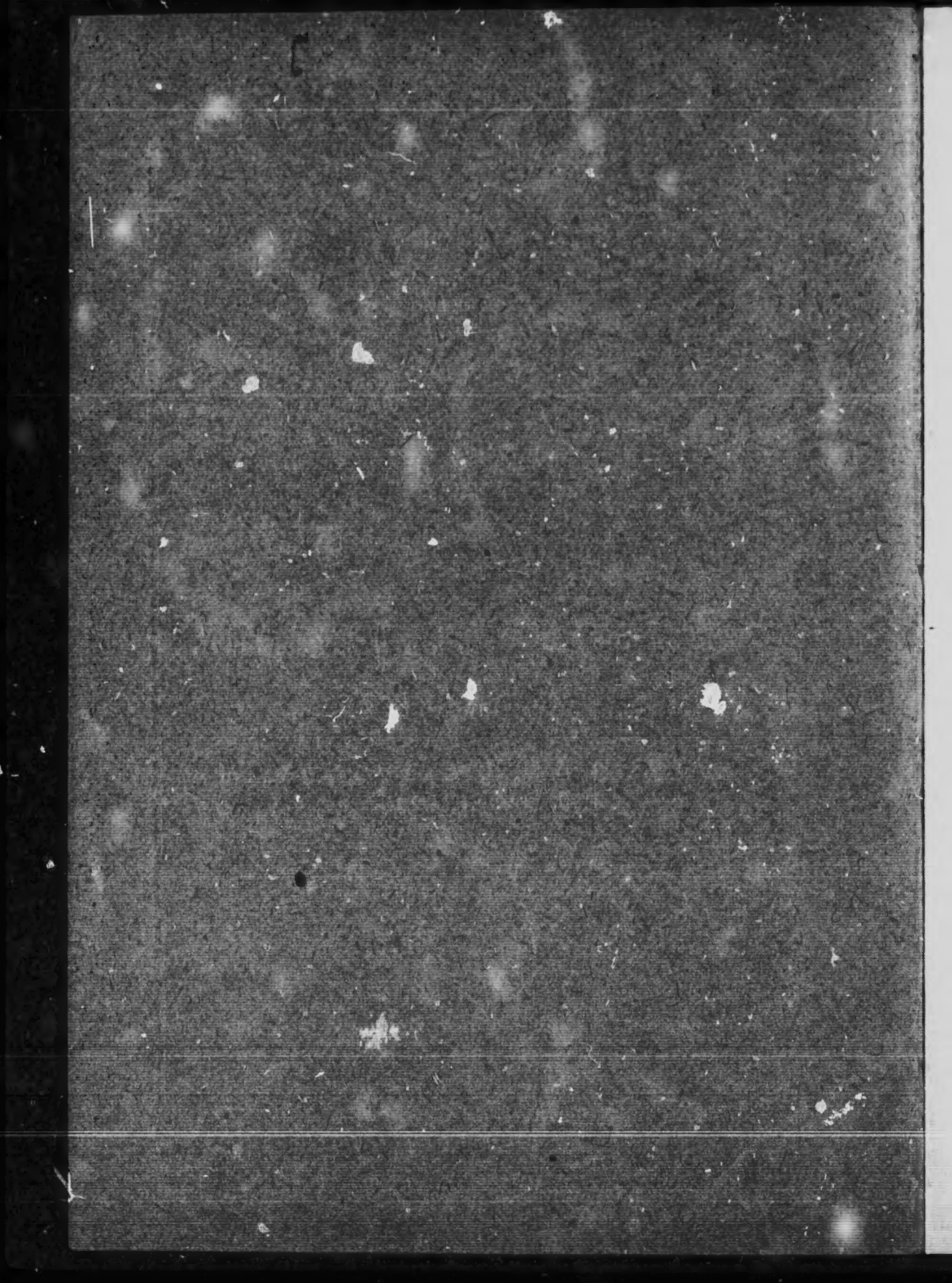
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These experiments were undertaken to determine on a suitable method for accurately estimating the amounts of arsenic, antimony, and tin in a mixture. Various processes were tried as recommended in different text-books on qualitative analysis, all of which had proved, in the authors' opinion, more or less unsatisfactory.

I.—The principles involved in the first method devised were the oxidation of the arsenic sulphide to arsenic acid, and the subsequent separation of the antimony sulphide from that of tin by digestion with tartaric acid. The process was thus carried out:—Known quantities of pure arsenic acid, tin chloride, and tartar emetic, were dissolved in water, with addition of sufficient hydrochloric acid (arsenic free) for complete solution. The whole was made up to a known volume, and aliquot parts taken for subsequent experiments. After reduction with sulphur dioxide the elements were precipitated as sulphides with sulphuretted hydrogen. It was found that precipitation occurred best in hot solution, not too strongly acid. The washed precipitates were evaporated to dryness with 15 c.c. of concentrated nitric acid, and the arsenic acid extracted by digestion in the water-bath with 100 c.c. of water. In the resulting filtrate, the arsenic was determined as $Mg_2As_2O_7$.

For the antimony, the residue from the last filtration was digested with a concentrated solution of tartaric acid for one hour, then filtered, and the residue washed, ignited, and weighed as SnO_2 .

To the solution of antimony oxide in tartaric acid were added hydrochloric acid and a considerable excess of water, and the antimony then precipitated as sulphide. Oxidation with nitric acid and subsequent ignition brought it into the state of oxide (Sb_2O_3), in which it was finally weighed.

Our results were as follow:—For each experiment, 20 c.c. of a solution were employed containing 0.0800 grm. of As_2O_3 , 0.0800 grm. of $SnCl_4$, and 0.0731 grm. of Sb_2O_3 .

and the amounts of arsenic, tin, and antimony found were calculated to the above.

—	I.	II.	Average.
	Grm.	Grm.	Grm.
As ₂ O ₃	0·0801	0·0799	0·0800
SnCl ₄	0·0804	0·0797	0·0801
Sb ₂ O ₄	0·0732	0·0723	0·0727

The digestion of the mixed oxides of antimony and tin is best carried on in a flask, with a glass tube and cork attached to prevent evaporation, sunk in a water bath.

II.—The method here employed was to separate the sulphides of antimony and tin by means of concentrated hydrochloric acid. The results of several experiments showed that an appreciable quantity of arsenious sulphide was dissolved by the hydrochloric acid when warmed, though the method of separation is advocated in most text-books.

In this Journal, 1898, 211—214, Messrs. J. & H. S. Pattinson showed that the addition of two-sevenths by volume of hydrogen sulphide water sufficed to prevent the solution of any of the arsenious sulphide, whilst the sulphides of antimony and tin dissolved readily. Nothing is said of the temperature at which this reaction took place. We have tried the effect of different concentrations of hydrochloric acid alone at different temperatures, and found the amounts of arsenious sulphide, which are dissolved, to vary very considerably. It is our intention to investigate the solubility of sulphide of arsenic under varying conditions of temperature, concentration, and relative masses of sulphide and acid; but as far as our present examination has gone, these results point to the method of separating the sulphides by hydrochloric acid as being unreliable.



